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भाग 4 चाँदी, ताँबा, कैडमियम और जस्ता निर्धारण की विद्युत अपघटनी पद्धति

Indian Standard

CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART 4 DETERMINATION OF SILVER, COPPER, CADMIUM AND ZINC — ELECTROLYTIC METHOD

ICS 77.120.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 2001

Price Group 1

Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee, MTD 28

FOREWORD

This Indian Standard (Part 4) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Anaylsis of Non-Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 2927 : 1975 'Specification for brazing alloys (*first revision*)'covers 22 grades of silver-copper brazing alloys. In order to determine correctly the composition of different copper-silver brazing alloys, method of analysis have been described in various parts of this standard.

The determination of silver and copper in silver-copper brazing alloys except BACuAg7 and BACuAg8 is covered in IS 4667(Part 1) : 1968 'Methods of chemical analysis of silver-copper brazing alloys: Part 1 Analysis of silver and copper'.

The determination of silver, copper and tin in BACuAg 7 and BACuAg 8 alloys are covered in IS 4667 (Part 2) : 1969 'Methods of chemical analysis of silver-copper brazing alloys: Part 2 Determination of silver, copper and tin'.

The determination of zinc by dimethylglyoxime method is covered in IS 4667 (Part 3) : 2001 'Chemical analysis of silver-copper brazing alloys: Part 3 Determination of nickel'.

This Part 4 covers the determination of silver, copper, cadmium and zinc by electrolytic method.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

AMENDMENT NO. 1 MAY 2002
TO
IS 4667 (PART 4) : 2001 CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART 4 DETERMINATION OF SILVER, COPPER, CADMIUM AND ZINC — ELECTROLYTIC METHOD

(*Cover page, Hindi title*) — Substitute the following for the existing:

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(MTD 28)

Reprography Unit, BIS, New Delhi, India

Indian Standard

CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART 4 DETERMINATION OF SILVER, COPPER CADMIUM AND ZINC — ELECTROLYTIC METHOD

1 SCOPE

This standard (Part 4) describes electroyltic method for determination of silver, copper, cadmium and zinc in silver-copper brazing alloys in the ranges given below:

Element	*Range* (Percent by mass)
Silver	9 - 93
Copper	7 - 53
Cadmium	15 - 22
Zinc	3 - 39

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2927 : 1975	Specification for brazing alloys (*first revision*)
4667 (Part 1) : 1968	Methods of chemical analysis of silver-copper brazing alloys: Part 1 Analysis of silver and copper

3 SAMPLING

The sample shall be drawn and prepared as per the method specified in IS 2927.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water shall be employed for the test.

5 DETERMINATION OF SILVER, COPPER, CADMIUM AND ZINC

5.1 Outline of the Method

The sample is dissolved in nitric acid and perchloric acid and silver, copper, cadmium and zinc are deposited electroytically at different currents and voltages.

5.2 Apparatus

The details of the apparatus for electrolysis are given in **5.2** of IS 4667 (Part 1).

5.3 Reagents

5.3.1 *Dilute Nitric Acid* — 1 : 1 (*v*/*v*).

5.3.2 *Perchloric Acid* — 72 percent (*v*/*v*).

5.3.3 *Ethanol* or *Methnol* — free from moisture.

5.3.4 *Sodium Hydroxide* — solid.

5.3.5 *Sodium Cyanide* — solid.

5.4 Procedure

5.4.1 *Dissolution of Sample*

Dissolve 0.5 to 1.0 g of sample in 5 ml of dilute nitric acid (1 : 1) and boil off nitrous fumes. Add 5 ml of perchloric acid (72 percent). Evaporate to fumes and heat till nitrate are decomposed. Cool and dilute to about 100 ml with distilled water.

5.4.2 *Determination of Silver*

5.4.2.1 Transfer the solution (**5.4.1**) into a 500-ml beaker. Insert the tared electrodes, cover the beaker with split watch glasses. Electrolyse the solution at 1.2 V (current approx. 0.25 amp) with constant stirring (about 200 rpm). Continue passing current till the voltage tends to rise and current to fall, indicating the completion of electrolysis.

5.4.2.2 As soon as the deposition is complete, lower the beaker slowly while washing the cathode with water without stopping the current. Preserve the electrolyte for next determination. Remove the cathode, rinse it with water and then dip in two successive baths of ethanol. Dry for 3 to 5 min in an oven at 105°C. Cool and weigh the cathode immediately.

5.4.2.3 Clean the cathode by dissolving in dilute nitric acid, dry and weigh.

5.4.2.4 Determine the weight of silver deposited (*A*) from the difference in mass of cathode obtained as in **5.4.2.2** and **5.4.2.3**.

5.4.2.5 *Calculation*

$$\text{Silver, percent by mass} = \frac{A}{B} \times 100$$

where

A = mass, in g, of silver deposited; and

B = mass, in g, of sample taken.

5.4.3 *Determination of Copper*

5.4.3.1 Insert the tared electrodes in the electrolyte solution preserved in **5.4.2.2**. Cover the beaker with split watch glasses and electrolyse the solution at 1.7 V. Add further 5 ml of perchloric acid and deposit copper with vigorous stirring (600 rpm) at 2.2 V (current opproximately 0.74 amp). Continue passing current for about 1 h till the solution becomes colourless, indicating complete deposition of copper. When the deposition is complete, reduce the voltage to 2 V before the stirring is stopped. Follow rest of the procedure specified in **5.4.2.2**. Clean the cathode by dissolving the deposited copper in dilute nitric and follow rest of the procedure specified in **5.4.2.3**.

5.4.3.2 *Calculation*

$$\text{Copper, percent by mass} = \frac{C}{D} \times 100$$

where

C = mass, in g, of copper deposited; and

D = mass, in g, of sample taken.

5.4.4 *Determination of Cadmium*

5.4.4.1 Insert the tared electrodes in the electrolyte solution preserved in **5.4.3.1**. Cover the beaker with split watch glass and electrolyse the solution at 3.4 V with rigorous stirring. When the deposition is complete, reduce the voltage, before the stirring is stopped. Follow rest of the procedure as specified in **5.4.2.2** and clean the cathode by dissolving the deposited cadmium in dilute nitric acid (1 : 1). Follow rest of the procedure as specified in **5.4.2.3**.

5.4.4.2 *Calculation*

$$\text{Cadmium, percent by mass} = \frac{E}{F} \times 100$$

where

E = mass, in g, of cadmium deposited; and

F = mass, in g, of sample taken.

5.4.5 *Determination of Zinc*

5.4.5.1 To the electrolyte solution preserved in **5.4.4.1**, add an excess of sodium hydroxide (*see* Note) and electrolyse zinc in the usual way at 6 to 7 V keeping current at 3 to 4 amp. Follow rest of the procedure specified in **5.4.2.2** and **5.4.2.3**.

NOTE — If any turbidity appears, clear it by the addition of a little sodium cyanide.

5.4.5.2 *Calculation*

$$\text{Zinc, percent by mass} = \frac{G}{H} \times 100$$

where

G = mass, in g, of zinc deposited; and

H = mass, in g, of sample taken.

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Amendments Issued Since Publication

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